

Sir:

PATENT APPLICATION Mo5457 MD-96-52-PU



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICATION OF **GROUP NO.:** 1711 KARL W. HAIDER ET AL R. A. SERGENT **EXAMINER:** SERIAL NUMBER: 09/474,114 FILED: DECEMBER 29, 1999 TITLE: HYDROPHOBIC LIGHT STABLE POLYURETHANE ELASTIMER WITH AECENED ABA 1 2 7013 ABA 1 2 7013 IMPROVED MECHANICAL **PROPERTIES**

DECLARATION UNDER 37 CFR 1.132

Assistant Commissioner for Patents Washington, D.C. 20231

I, Karl W. Haider, of Hurricane, West Virginia, declare as follows:

- I studied chemistry at Ohio State University and obtained a Ph.D 1. degree in 1989.
- I am employed by Bayer Polymers LLC in South Charleston, West 2. Virginia as a Principal Scientist.
- I am one of the named inventors of the above-identified United States 3. patent application.
- I performed or supervised the experiments described in the aboveidentified United States patent application.

5. The OH terminated homopolymer of butadiene made in Example 1 of the above-identified United States patent application had the following physical properties:

Equivalent weight 1300

 M_n

2600

Viscosity

800 cP

OH number

52.0 mg KOH/g

- 6. I further declare that I am one of the named inventors of United States Patent Number 6,166,166, and I unequivocally declare that I, along with my coinventors, conceived of or invented the subject matter disclosed therein.
- 7. The undersigned declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the above-identified United States patent application or any patent issuing therefrom.

Signed at Hurican, WU, this 25th day of February, 2003.

Karl W. Haider

/jme/JM/JM0114

EXHIBIT 1

find applications as automotive cushions, carpet underlay, furniture, seating,

Rigid foams are based primarily on polyfunctional, low molecular weight and bedding. alcohols and amines. Most global applications conventionally use polymeric isocyanates, TDI, or an undistilled grade of mixed TDI isomers. TDI prepolymers which have hydroxyl and isocyanate groups have been marketed as a low vapor pressure alternative to undistilled TDI. Density reduction is effected via the addition of chlorof luorocarbons, low molecular weight alkanes, or via the in situ generation of carbon dioxide. The resultant closed cell foams find applications as insulators in construction, appliance, transportation, pipeline, and tank

Similarly, polyisocyanurate (PIR) rigid foams are based on PMDI and polyester or polyether polyols (and blends thereof). The cross-link density, aroend uses. matic content, cell wall thickness, and polymer distribution are known to have a pronounced effect on the overall performance of the foam. Polyisocyanurate foams are used extensively in industrial applications having service temperature ranges from -200 to 150°C. These foams are known to provide efficient thermal insulation characteristics and structural integrity to a variety of composite applications. Apart from roofing and sheathing, PIR foams are used in garage doors, building panels, and foaming applications. They can also be supplied in the form of boardstock for fabrication into sheets, pipe covering, and

Adhesives and coatings formulations utilize both MDI and TDI along with a variety of polyether and polyester polyols. The largest segment of the business other shapes. is the one-part or moisture-cured approach, which is heavily reliant on prepolymers. Optionally, blocked isocyanate technology may be used to provide systems having an extremely long shelf life. Suitable blocking agents include phenols, diethyl malonate [105-53-3], acetone oxime [127-06-0], and ϵ -caprolactam [105-60-2]. Typical applications include flexible film packaging and wood furniture assembly. Two-part reactive adhesives employ a mixture of pure isocyanates and prepolymers and are primarily used for industrial product assembly and

Elastomers are segmented block copolymers. They employ a mixture of pure commercial construction. MDI-based isocyanates and prepolymers. The hard blocks consist of high melting MDI-glycol units which aggregate via hydrogen bonding to provide a high degree of virtual cross-linking. Suitable glycols include ethylene glycol [107-21-1], butanediols, and hexanediols. The soft block consists of high molecular weight poly(alkylene oxide) polyols. These materials are noted for their resiliency, abrasion resistance, solvent resistance, and the high level of tensile, tear, and elongation performance. Applications include shoe soles, wheels, rollers, belts, gaskets, and thermoplastic polyurethanes. Methylene diphenyl diisocyanate (MDI) is used extensively in the production of Spandex fibers. Spandex is used in foundation garments and swimwear (see FIBERS, ELASTOMERIC). Typical demand for various applications is presented in Table 7.

Aliphatic Isocyanates. Aliphatic diisocyanates have traditionally commanded a premium price because the aliphatic amine precursors are more expensive than aromatic diamines. They are most commonly used in applicaC) iı C ť

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HYDROXYL-TERMINATED POLYBUTADIENE TELECHELIC POLYMER (HTPB): BINDER FOR SOLID ROCKET PROPELLANTS

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ABSTRACT

This paper presents a review of hydroxyl-terminated polybutadiene telechelic polymer (HTPB) with emphasis on its preparation, properties, end group modifications, hydrogenation, role as polyurethane precursors and binders for solid rocket propellants.

1. INTRODUCTION

Telechelic polymers are of great interest, mainly because they provide the means for further reactions to give modified polymers with controlled properties. The term telechelic (from the Greek tele: far, distant and chelos: claw) has been proposed by Uraneck, Hsieh and Buck' to describe the polymers possessing two functional terminal gr ups. Telechelic polybutadienes with different functional groups have been prepared, as shown in Table I, but hydr xyl-terminated polybutadienes (HTPB) have been the subject of much developmental work because of their wide applications, such as, binders in solid rocket propellants, expl sives, adhesives, sealants, coating materials, etc.

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TABLE I
TRLECHELIC POLYBUTADIRNES WITH DIPPERENT FUNCTIONAL GROUPS

Initiator	Terminal group	Reference
2,2'-Azobis(isobutyronitrile) Xanthogenate disulphide Xanthogen disulphide Dilithium isoprene/S Glutaric acid peroxide 4,4'-Azobis(4-cyanovaleric acid) Cyclohexanone peroxide/ferrous salt Ethyl-2,2'-azobis(isobutyrate) 4-t-Butyl-azo-4-cyanovalery) chloride Aromatic acid diacyl peroxide Carbon tetrabromide	Nitrile Thiol Thiol Thiol Carboxyl Carboxyl Carboxyl Carboethoxy Ester with aliphatic azo group Chloro or bromo Bromo	9 9 10 11 12 13 14 15, 16 17

Two reviews have been written previously on HTPB. One is related to its synthesis, physical and chemical properties, and propellants' while the other describes the recent developments and applications.

II. PREPARATION

Free radical and anionic polymerizations are the two major routes employed for the synthesis of HTPB. However, it has been reported²¹ that the polymers obtained by the polymerization of 1,3-butadiene with azoinitiators have narrow molecular weight distribution compared to those obtained by using hydrogen peroxide as an initiator. On the other hand, the polymers prepared by anionic polymerization are superior in properties to those obtained by free radical polymerization, but this route is expensive and used for specialized applications only. Table II²²⁻²⁸ shows some hydroxyl-terminated poly butadienes which are available commercially. Stephens et al.²⁷ reported the synthesis of low molecular weight hydroxyltadiene, as shown in Figure 1.

A. FREE RADICAL MECHANISM

Free radical polymerization is the most commonly used method to synthesize the hydroxylterminated polybutadienes. Chain termination in this kind of polymerization must be a combination and it occurs almost exclusively by combination in the case of butadiene. It is important to recognize how the various types of monomer units are produced during radical polymerization of butadiene.

TABLE II
COMMERCIALLY AVAILABLE HTPB

Trade	TO STATE OF THE PARTY AND THE	BLE HTPB	
Trade name R 15, R 45 M	Source	Method of preparation	Reference
Butarez HT Poly bd R 45 HT and R 45 M Poly bd R 45 HT	Sinclair Philips Sartomer Co. Idemitsu Pêtr. Co.	Anionic Free radical Free radical	22, 23 24 25 26

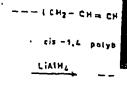


Fig.

Attack of a radical resonance and rotation monomer molecule by if it reacts as (D), a ci the form (B). In radic: are not affected by the or the conversion, but performed²⁰. The 1,4 the cis as the reactior with butadiene is about

Kalaus et al. 31 esti 20% cis, 60% trans-1, viscosity of carboxylmay be estimated for viscosity on going fr. Thus, variations in the trans ratio, dubtless with viscosity. Viscopolymer. The function reproducible. 35 The function two because the emulsion polymerized and about 20% 1,2-e.





FIG. 2. - Various t

n nterminal OH groups.³⁴ Random OH functions are quite undesirable for application in propellant binders.

1. Hydrogen peroxide initiation. — Several scientists have used hydrogen peroxide as an initiator for the polymerization of butadiene to obtain HTPB. M-61 This free radical addition polymerization usually takes place at a temperature of about 100-200°C, preferably about 100-150°C. The reaction preferably takes place in a mutual solvent system; that is, one which dissolves both the diene monomer and the hydrogen peroxide. Suitable solvents are isopropanol, acetone, methanol, sec-butanol, n-butanol, n-propanol and like alcohols having two to about twelve carbon atoms. The hydrogen peroxide-solvent system is found to supply hydroxyl groups and the catalytic and solvent effects needed to produce the polymer of desired chemical and physical characteristics. The alcohol is used in an amount suitable to promote adequately rapid but controllable polymerization of the monomer in the solution to f rm the polymer. The alcohol must be free of any group which would interfere with the production of the butadiene polymer. Propanol or isopropanol are the preferred solvents. The amount of hydrogen peroxide used is crucial because it determines the molecular weight of the final product.

Isaacson and Young⁴⁸ obtained the HTPB of an average molecular weight 3000-3500 by reacting 100 parts of 1,3-butadiene and 6 parts of hydrogen peroxide whereas the HTPB of an average molecular weight 2200-2500 was obtained when 100 parts of butadiene were mad to react with 10 parts of hydrogen peroxide. The product is found to have mono, in and tri-functional molecules.⁵²

Besides the cleavage of hydrogen peroxide by strong acids and heat, it can also be cleaved by oxidation reduction (redox) initiator, i.e., ferrous ion/hydrogen peroxide, 50 as shown in Equation (1).

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + \cdot OH$$
 (1)

This redox system is often used in the presence of alcohols like methanol or isopropanol. There are two main side reactions associated with this system. The ferric ions may be reduced by hydrogen peroxide, as shown in Equation (2).

$$Fe^{3+} + H_2O_2 \rightarrow Fe^{+2} + H^+ + HOO$$
. (2)

Due to the high reactivity of \cdot OH radicals an H abstraction from the solvent can also take place as shown in Equation (3)

$$HO \cdot + CH_3OH \rightarrow H_2O + \cdot CH_2OH$$

The high reactivity of the hydroxyl radical will also give rise to other transfer reactions if the conditions are favorable.²⁸

A simplified structure of HTPB, 25 obtained by hydrogen peroxide radical polymerization of butadiene, is shown in Figure 3. The terminal hydroxyl groups are primary and predom-

Fig. 3. —Structure of HTPB obtained by $\rm H_2O_2$ radical polymerization.

inantly of the allylic type sation polymerizations a

2. Azoinitiators, because of their low proj with primary or second: Azoinitiat rs with ts

(a) Strecker type sy appropriate keto alcohcyanide, followed by th

(b) Pinner type synt available 2,2'-az bisisot ence of dry hydrogen cl

Different types of hy in the synth sis of HTl report the synthesis of free radical polymeriza and 5,5'-azobis(5-cyanc tadiene prep lymers at the cure reaction of suc to pot life and casting. 6' di(4-hydroxybutyl)-2,2 pentanol) has been des

Of the az initiators, initiation by this initia and very good function to 3.8, for butadiene of catalyst addition 31.84 m well as for narrow mo dergoes first-order decor transfer reactions, a used with this initiate to have low chain transfer tra

Szwarc^{68,67} was the later followed by differ has reviewed in detail

> Diethyl-2,2'-a: Di(4-hydroxy) Di(3-hydroxy) Di(2-hydroxy) Di(2-hydroxy) 5,5'-Azobis(5-4,4'-Azobis(4-

By reduc

CH 1 | CH3

ns f ani nic polyth suitable el ctroionally substituted tal techniques for the equipment and of impurities that tol is better in this nality values to be eneral be narrower

d in Table IV.
nonpolar solvent,
however, to obtain
ess of the metal is
ctures of the poly-

e lithium initiated

by reaction with obtained. Ethylene stly association of de is less reactive, que) and secondary

rahydropyran and me and conversion minated polymers I molecular weight

TABLE V

MICROSTRUCTURE OF ALKALI METAL CATALYZED POLYBUTADIENES⁸¹

Catalyst	% Cis-1,4	% Trans-1,4	% Vinyl-1,2
Lithium	35	52	13
Sodium	10	25	65
Potassium	15	40 .	45
Rubidium	7	31 .	62
Cesium	6	35	59

distributions and also that the procedure is not handicapped by the gelation problems, which are normally encountered during the termination of dilithiopolymers with electrophilic reagents.

III. PREPOLYMER PROPERTIES

Characterization of HTPB can be carried out by taking into consideration the following properties: (a) Molecular weight and molecular weight distribution; (b) Equivalent weight; (c) Microstructure; (d) Functionality and functionality distribution; (e) Moisture content; (f) Nonvolatile material; (g) Hydroxyl value, meq/g; (h) Hydroxyl number, mg KOH/g; (i) Iodine number; (j) Density; (k) Refractive index; and (l) Heat of combustion.

Table VII shows some properties of commercially obtained HTPBs. The density of average samples of HTPB is 0.895. The index of refraction is 1.515 ± 0.003 . The heat of combustion is 18,100 BTU/lb. The properties important to a formulation chemist are molecular weight and molecular weight distribution, microstructure, and reactive end group functionality. These properties will now be discussed in some detail.

A. MOLECULAR WEIGHT AND MOLECULAR WEIGHT DISTRIBUTION

Molecular weight and molecular weight distributions $(\overline{M}_w/\overline{M}_n)$ are of great importance in telechelic polymers. It has been reported that the telechelic polymers characterized by narrow molecular weight distributions tend to cure faster and show a lesser tendency towards net-

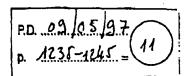
TABLE VI
POLYMERIZATION OF BUTADIENE WITH LITHIUM⁸¹

	Butadiene	utadiene Li°	Solvent		Ø	C-1			/
No.			Туре	Conc.	Temp. (°C)	Gel %	% Cis-1,4	% Trans-1,4	% Vinyl-1,2
1	100	0.1	None		60	1	29.1	57.2	13.6
2	100	0.3	None		50	4	29.0	58.2	12.8
3	100	0.3	None		60	11	30.0	57.0	13.0
4	100	0.3	None		30	0	31.5	58.2	10.3
5	100	0.3	1,E.	68.0	40	1	29.0	60.5	10.4
6	100	0.3	ΡĒ	68.0	50	2	28.1	60.9	11.0
7	100	0.3	PE	68.0	50	15	32.0	57.3	10.6
8	100	0.3	PE	68.0	70	14	30.0	58.0	11.9
9	100	0.5 `	PE	68.0	40	2.8	28.9	60.3	10.8

^a Lithium metal as 35% dispersion in petroleum.

^b All concentrations are in parts by weight.

Petroleum ether.



XP-000802660

Tensile Property of Modified Hydroxyl-Terminated Polybutadiene-Based Polyurethanes

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ABSTRACT: Modified hydroxyl-terminated polybutadiene (HTPB)-based polyurethanes (PUs) were prepared by three different processes: crosslinkage of the soft segment, complexation of the hard segment, and the interpenetrating network (IPN). Crosslinked PU films were prepared by the addition of a crosslinking agent of divinylbenzene (DVB) to the 4.4 '-dicyclohexylmethane diisocyanate (H₁₂MDI) and 1,4-butanediol (1,4-BD) based PUs. The ionic polymer was prepared by adding N-methyldiethanolamine (MDEA), which possesses tertiary amine used as chain extender, to the HTPB-H12MDI-based PUs and then complexed by cupric chloride with MDEA. An IPN was formed by the introduction of 4-vinylayridine (4-VP) to the benzoyl peroxide (BPO)crosslinked HTPB-H12MDI-1,4BD-based PUs. FTIR was utilized to identify the segregation between hard and soft segments and structure change, which affects the tensile properties. The change of the glass transition temperature was detected by DSC, which can be used to manifest the modified PUs. Thermal decomposition behaviors conducted by TGA were used to investigate the formation of an IPN. The hydrogen-bonding index (HBI), frequency difference, and shift as a measure of the phase segregation and the average strength of the interpolymer hydrogen bonds were utilized to study the intermolecular interaction and tensile property of the prepared PUs. The effect of the hard-segment content, DVB content, amount of cupric ion, and 4-VP content on the tensile property were investigated. The stress was largely increased by the modification of PUs, while the strain still remains an appreciable value compared with that of unmodified PUs. 6 1997 John Wiley & Sons, Inc. J Appl Polym Sci 84: 1235-1245, 1997

INTRODUCTION

Polyurethane (PU) materials possess very attractive bulk mechanical properties due to the presence of phase-separated structures. Its applications are varied, such as for medical implants, membranes, adhesives, and coatings. Superior hydrolytic stabilities and high mechanical property performances of HTPB show surprising utility in many fields.1-1 HTPB-based PUs have been used for gas separation research because of their

low-temperature flexibility and high segregation between hard and soft segments. 4.5 Pervaporation separation of an ethanol/water mixture was also studied with HTPB-based PUs due to the high swelling behavior of the HTPB soft segment with ethanol.

Many reports have dealt with the structure and mechanical properties of HTPB-based PUs. ---But the HTPB-based PUs generally exhibit inferior mechanical properties compared to the conventional PUs.20 The purpose of this study was to prepare modified HTPB-based PUs for the improvement of mechanical properties. The modification was carried by the crosslinkage of the

Correspondence to: S.-L. Huang. 0 1997 John Wiley & Sons. Inc. CCC 0021-8995/97/061235-11

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HTPB soft segment, complexation of the hard segment, and the interpenetrating network (IPN). From the reported literature, the crosslinkage of the PU polymer was generally obtained by the introduction of diol and triol mixtures to the PU prepolymer.21,22 Yoshikawa et al.23 studied the crosslinking of polybutadiene (PB) membranes arising from chemical reactions with the crosslinking agent of bis(1-methyl-1-phenyl-ethyl)peroxide at the sites of unsaturation in the polymer chain. Sun and Ruckenstein prepared divinylbenzene (DVB)-crosslinked styrene/butadiene/styrene (SBS) and styrene/ethylene-butylene/styrene (SEBS) for alcohol permselective membranes, while PU ionomers were prepared by incorporating a chain extender containing tertiary amines and then reacting with alkyl halides 23 or glycolic acid. 26 Others are formed by the use of sulfanate containing a chain extender 27 or by the addition of LiClO, to the PUs.28.29

This study attempted to prepare crosslinked HTPB-based PUs by using DVB to crosslink between hard and soft segments of butadiene. The PU complexes were obtained by adding cupric chloride to the HTPB-H12MDI-MDEA-based PU solutions, while IPNs were prepared by adding 4-VP to the BPO-crosslinked HTPB-H12MDI-1,4BD-based PUs. The effect of the hard-segment content, cupric chloride content, and 4-VP content on the tensile property was studied. Hydrogenbonding index (HBI) values for indication of phase segregation were measured by FTIR. The measurements of DSC and FTIR were used to identify the degree of crosslinking and complexation and, hence, to correlate them with the tensile property. HBI values, the IR frequency difference, the frequency shiftment, and the glass transition temperature were used for the identification of phase homogeneity and for the effect on the tensile property.

EXPERIMENTAL

Materials

The chemicals used in this study were 4,4'-dicyclohexylmethane diisocyanate (H₁₂MDI Desmodur W of Mobay Co.), hydroxyl-terminated polybutadiene (HTPB, equivalent weight 1333 g, R-45M of ARCO Co.), 1,4-butanediol (1,4-BD), and N-methyldiethanolamine (MDEA) as chain extenders and dibutyltin dilaurate (DBTDL) was used as a catalyst. Dimethylformamide (DMF)

and toluene were used as solvents for the preparation of the PU solution. Benzoyl peroxide (BPO) was used as an initiator for the preparation of divinylbenzene (DVB)-crosslinked PUs and as a crosslinking agent to the PU solution of 4-VP/PU IPNs. The 4-VP monomer was purified by vacuum distillation.

Preparation of Polyurethane Membrane

The two-stage PUs were polymerized first by a —NCO-terminated prepolymer and then chain-extended with 1,4-BD (or MDEA) under 25 wt % solid content after the theoretical —NCO content was reached. It was diluted to a 15-20 wt % solid content after the chain-extended reaction proceeded for 30 min. The reaction was terminated as the —NCO groups were completely consumed, as confirmed by the disappearance of the infrared absorption at 2280 cm⁻¹.

Uncrosslinked PU films were prepared by pouring the solution mixture onto a glass plate to a thickness of 600 µm. The solvent in the casting solution was evaporated by degassing at 65°C for 24 h. The dried PU films were peeled from the plate after it had been immersed in deionized water for several hours. The PU films were dried in a desiccator and stored at a relative humidity of 50% and 25°C for 5 days before testing.

Crosslinked films were initially prepared by adding 0.5 wt % (based on solid PU content) of BPO to the HTPB-H₁₂MDI-1,4BD-based PU solution (15 wt % solid content) in the closed vessel at room temperature for 30 min. Then. 1.0, 2.0, 3.0, and 5.0 wt % of DVB (based on solid PU content) were added to the above PU solution at 65°C for 30 min, respectively, and then cooled to room temperature immediately. All the following film-preparation steps were the same as those of uncrosslinked films.

Complexed films were prepared by adding cupric chloride to the HTPB-H₁₂MDI-MDEA. based PU solutions. Cupric chloride with MDEA of 1/3 or 1/4 mol ratio content, was added to the three different PU compositions, respectively. Cupric ion forms a complex with tertiary amine of MDEA by ion-dipole interaction. The dried ionized films were peeled from the plate after it had been put into refrigerator and into the frozen state for several hours. All these films were then put in an oven under a vacuum at 70°C for 48 h for further degassing of the solvent residue. Finally, the samples were kept under a vacuum at room

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** TOTAL PAGE.08 **

EXHIBIT 3

Product Bulletin

Poly bd® Resins

Hydroxyl Terminated Polybutadiene Resins and Derivatives

SARTOMER



502 Thomas Jones Way, Exton, Pennsylvania 19341 610-363-4100 • 1-800-SARTOMER • www.sartomer.com

Poly bd® R-45HTLO Resin

HYDROXYL TERMINATED POLYBUTADIENE RESIN

DESCRIPTION

Poly bd® R-45HTLO resin is a liquid hydroxyl terminated polymer of butadiene with a number average molecular weight of approximately 2800. Poly bd® resins have primary, allylic alcohol groups that exhibit high reactivity in either condensation polymerization reactions or the preparation of derivatives. The degree of polymerization is approximately 50 for the R-45HTLO. Hydroxyl functionality is typically in the 2.4 – 2.6 range for R-45HTLO. Poly bd® R-45HTLO is regulated by the United States Department of Commerce and may not be exported without a license from that organization.

PRODUCT HIGHLIGHTS

Hydrophobicity
Reactive hydroxyl groups
Low glass transition temperature
Miscibility with asphalt
Low color, high clarity

PERFORMANCE PROPERTIES

Hydrolytic stability
Low temperature Flexibility
Low moisture permeability
Resistance to aqueous acids and bases
Excellent adhesion to a variety of substrates
Electrical insulation properties

SUGGESTED APPLICATIONS

Potting and encapsulation
Adhesives
Sealants
Binders
Waterproof coatings and membranes

Poly bd® R-45HTLO Resin TYPICAL PHYSICAL AND CHEMICAL PROPERTIES

Nonvolatile Material, wt%	99.9
Viscosity, mPa·s @ 23°C	8000
Viscosity, mPa·s @ 30°C	5000
Hydroxyl Number, mg KOH/g	47.1
Hydroxyl Value, meq/g	0.84
Hydroxyl Functionality	2.4-2.6
Molecular Weight, M	2800
Polydispersity, M_/M_	2.5
Water, wt%	0.02
Specific Gravity @ 23°C	0.901
Iodine Number, g/100g	400
Glass Transition Temp. (Tg), °C	-75
Solubility, g/100 ml of solvent @ 25°C	
Mineral Spirits	>50
Toluene	>50
Chloroform	>50
Methyl Ethyl Ketone	>50
Ethyl Acetate	>50
Acetone	<10(1)
Hexane	>50
Aromatic 100	>50
Isopropanol	<10(1)
• •	

(1) Cloudy: 5% solution also cloudy

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